

Patent Application
Docket No. USF-221XT
Serial No. 09/763,419

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Examiner : Arlen Soderquist
Art Unit : 1743
Applicants : Abdul Malik, Dongxin Wang
Serial No. : 09/763,419
Filed : July 19, 2001
Conf. No. : 6244
For : Capillary Column and Method of Making

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION OF ABDUL MALIK, Ph.D., UNDER 37 CFR 1.132

Sir:

I, ABDUL MALIK, hereby declare:

THAT, I am a co-inventor of the subject matter claimed in U.S. patent application Serial No. 09/763,419 (hereinafter the '419 application);

THAT, a copy of my curriculum vitae is attached hereto as Exhibit A;

THAT, I have reviewed the Office Action mailed February 11, 2005, in the '419 application along with the references cited therein;

THAT, by virtue of my employment, my educational background, my research, my participation in authoring and reviewing articles and publications, my attendance at and participation in conferences and seminars, and my personal correspondence with others in the

field, I am aware of the level of skill of the ordinary artisan in the field of capillary chromatography and preparations of separation columns.

And, being thus duly qualified, do further declare as follows:

I. The subject invention is based on gas chromatographic columns having a deactivated column coating, wherein the deactivated coating also functions as the stationary phase of the column. One of the features of the coating is that it is deactivated at both the column surface and within the coating itself. The subject invention also includes methods of preparing these columns.

II. The Office Action states that the specification lacks written description supporting the exclusion of cross-linked organic ligands. As a co-inventor and expert in this field, the undersigned submits that a skilled artisan would understand that the organic ligands in the method claims, or the organic-inorganic composites in the column claims, are not cross-linked when reading the specification in view of the prior art. First, the specification includes several schemes that illustrate the deactivation of the column at the inner surface and embodiments for the formation of the columns and methods of the claimed invention. A skilled artisan viewing methods to prepare GC stationary phase coatings that do use cross-linking reagents would understand that the sol-gel coating of the claimed invention could not possess a cross-linked organic ligand. Second, the specification itself discusses the advantages of the claimed invention, one of which is its lack of free radical cross-linking reactions (page 28, line 14). The column would have no mechanism by which an organic polymer could cross-link. Thus, the skilled artisan would understand that the columns are prepared without cross-linking the organic ligands in the column coating.

III. The Office Action also states that the columns and methods of preparing the columns are obvious over the teachings of the Hayes *et al.* article (hereinafter 'Hayes *et al.* 1997') in view of Ogden *et al.* (1986) and Sumpter *et al.* (1990), and optionally Hayes *et al.* (hereinafter 'Hayes *et al.* 1996') and Wang *et al.* (1996). As an initial matter, the undersigned notes that he is a co-author of the Hayes *et al.* 1997 reference.

The Hayes *et al.* 1997 article fails to teach or suggest the claimed columns or methods of preparing the same. Although it possesses sol-gel coating on a capillary electrophoresis (CE)

column, its sol-gel coating is inapplicable to columns of the claimed invention for the following reasons. First, the Hayes *et al.* 1997 coatings possess highly cross-linked polymers to prevent the analyte molecules from interacting with the solute, thereby reducing separation efficiency. Second, the skilled artisan is not motivated to apply the Hayes *et al.* 1997 sol-gel coating to the gas chromatographic (GC) columns of the claimed invention because the highly cross-linked polymer coatings of Hayes *et al.* 1997 would result in poor solute diffusion and mass transfer of the analyte molecules.

Third, the CE column is not used to separate moieties chromatographically. By its definition, a CE column possess only a mobile phase and takes advantage of migration rates of ionic solutes in a liquid mobile phase environment when they are placed in an electric field. When these solutes are exposed to the inner surface of the column, any resulting interactions reduce the column efficiency. Any sol-gel coating applied to a CE column does not represent a stationary phase where separation of ionic analytes takes place because of differences in their electrophoretic migration rates in an electric field. As noted above, the sol-gel coating is applied only to prevent unwanted interactions between the solutes and the inner surface of the capillary. Fourth, the sol-gel coating of the Hayes *et al.* 1997 columns contains a highly cross-linked organic ligand. In contrast, the deactivated sol-gel stationary-phase coating of the claimed invention contains a chemically bonded non-crosslinked ligand.

The Ogden *et al.* article fails to remedy the deficiencies of Hayes *et al.* 1997. For one, the skilled artisan would not combine the teachings of Hayes *et al.* 1997 with the Ogden *et al.* article because the individual columns rely on two distinct separation theories. Whereas Hayes *et al.* 1997 relies on the migration rates of various ionic solutes in a liquid mobile phase environment, Ogden *et al.* uses its conventional (i.e., nonsol-gel) coating as a stationary phase. As noted above, Hayes *et al.* 1997 fails to even have a stationary phase within its column.

Additionally, the combined teachings of Hayes *et al.* 1997 and Ogden *et al.* do not teach or suggest the claimed invention. As noted above, the claimed invention does not possess cross-linked elements. Both Hayes *et al.* 1997 and Ogden *et al.* both teach cross-linked coatings of their individual columns (Hayes for sol-gel columns and Ogden for nonsol-gel columns). Additionally, the Ogden *et al.* article discloses a method for GC column preparation where deactivation takes place only at the surface of the capillary (pages 16-7). In contrast, the claimed

column, advantageously, possesses a deactivated sol-gel coating, which encompasses both the deactivation at the tube inner surface and throughout the coating.

Regarding the methods of the claimed invention, the Ogden *et al.* article relies on a multi-step approach in the preparation of its columns, wherein the deactivation step takes place separately from the immobilization step (pages 11-3). In contrast, the stationary phase of the claimed invention is created, coated, immobilized, and deactivated in one step.

Again, the skilled artisan is unlikely to combine the teachings of Sumpter *et al.* with Hayes *et al.* 1997. Sumpter *et al.*, similar to Ogden *et al.* discussed above, pertains to chromatographic columns (gas and supercritical fluid). The skilled artisan has no motivation to combine the teachings of a separation column based on chromatography with a separation column based on migration rates of ionic solutes in a liquid mobile phase environment. As noted above, sol-gel coatings in chromatographic columns are used as a stationary phase whose differential interactions with the analytes lead to separation. In contrast, the sol-gel coating in Hayes *et al.* 1997 is used to prevent interactions between the column and the solute.

Initially, even if the references were properly combinable, the combination of Hayes *et al.* 1997 in view of Sumpter *et al.* or the combination of Hayes *et al.* 1997 in view of Ogden *et al.* and Sumpter *et al.* fails to teach each and every element of the claimed invention. Regarding the method claims, the combination of references fails to teach a preparation of a sol-gel coating wherein the creation, the coating, the immobilization, and the deactivation take place all in one step. Specifically, the methods disclosed in the cited references are multi-step preparations.

Regarding the column itself, the combined references again fail to teach a column with a deactivated sol-gel stationary phase with a non-crosslinked organic ligand. As noted above, Hayes *et al.* 1997 teach the creation of a highly cross-linked sol-gel coating (which is not a stationary phase) within a capillary; the Ogden *et al.* and Sumpter *et al.* only teach deactivation at their respective column surfaces and/or cross-linking of a non-sol-gel stationary phase coating. Thus, the combination of references fails to teach or suggest a GC column with a sol-gel stationary phase coating (with a non-crosslinked organic ligand) that is deactivated at both the capillary inner surface and within the coating.

The Wang *et al.* reference also fails to remedy the short-comings of Hayes *et al.* 1997. The undersigned is also a co-author of the Wang *et al.* reference. For one, the Wang *et al.*

reference is directed to a non-aqueous preparation for a sol-gel coated GC column. In contrast, the Hayes *et al.* 1997 column is directed to an aqueous preparation for a sol-gel coated CE column. Wang *et al.* teaches against aqueous preparation of GC columns (page 507). Accordingly, a skilled artisan is unlikely to combine the teachings from a non-aqueous GC column with the teachings of an aqueous CE column to arrive at the claimed invention. Moreover, the skilled artisan understands that due to the innate differences in aqueous prepped GC columns and non-aqueous-prepped columns, the resulting column perform inefficiently, if at all (see, for example, Figure 2 from Wang *et al.* vs. Figures 31 and 36 of the subject application). In contrast, the columns of the claimed invention unexpectedly show highly efficient separations represented by narrow peaks that are narrow without the tailing exhibited by the Wang *et al.* column. Evidence supporting this superior separation column can be found throughout the instant application including, for example, Figures 7-39.

The Hayes *et al.* 1996 reference also fails to remedy the shortcomings of the Hayes *et al.* 1997 reference. The undersigned is also a co-author of this reference. First, Hayes *et al.* 1996 fails to suggest or teach a gas chromatography column having the deactivated surface coating of the claimed invention. The teachings of Hayes *et al.* 1996 apply to a CE column (page 497, lines 32-33). As noted above, the coating in a CE column is used to prevent unwanted interactions between the analytic and the column surface. The CE coating does not function as a stationary phase and fails to provide molecular level interactions responsible for chromatographic separation. Moreover, Hayes *et al.* 1996 only discusses sol-gel techniques as they have been applied to capillary electrochromatography, capillary electrophoresis, and open tubular liquid chromatography (page 500, lines 4-7). It provides no suggestion that a sol-gel coating could be applied to a GC column to separate analytes in a gaseous mobile phase environment.

Second, Hayes *et al.* 1996 fails to suggest that the coating could be prepared in the presence of water. The preparation steps explicitly avoid the use of water or even components having hydrated water molecules. The only disclosure for an aqueous preparation is with respect to a packed column. In contrast, the claimed invention pertains to coated columns. There is no need for a packing in the sol-gel columns of the claimed invention prepared by using an aqueous sol-gel process. Thus, the deficiencies of Hayes *et al.* 1997 are not remedied by the teachings of Hayes *et al.* 1996.

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The undersigned declare further that all statements made herein of his or her own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or of any patent issuing thereon.

Further declarant sayeth naught.

Signed:

Abdul Malik
By: Abdul Malik

Date:

06/09/2005

Exhibit A: Curriculum Vitae of Dr. Abdul Malik